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ROLE OF Hf AND Zr IN THE HYDROGEN EMBRITTLEMENT OF TA AND CD ALLOYS

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TECHNICAL PAPER proposed for presentation at Conference on Hydrogen in Metals: Effects on Properties, Selection, and Design sponsored by the American Society For Metals Champion, Pennsylvania, September 23-27, 1973 ROLE OF HF AND Zr IN THE HYDROGEN

EMBRITTLEMENT OF Ta AND Cb ALLOYS

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INTRODUCTION 44

Alloys of Ta and Cb are of interest as containment material for alkali liquid metals. In the presence of oxygen contamination concentrated at grain boundaries in such alloys as Ta-10W, severe corrosion results due to preferential attack of the high oxygen content regions by alkali metals. Because of this corrosion problem, additions of reactive elements such as Hf or Zr are made to Ta and Cb alloys to getter the oxygen. It has been shown (1) for a Ta alloy, T-111 (Ta-8W-2Hf), that corrosion was essentially nil after aging for several thousand hours in an alkali metal environment. However, recent results have indicated (2) that long term aging of T-111 near 1040°C (a proposed use temperature for this alloy) increases its sensitivity to hydrogen embrittlement during subsequent room temperature handling and testing in the presence of moist air.

The purpose of this paper is to further characterize the hydrogen embrittlement of aged T-III and similar Ta and Cb alloys and to describe the mechanisms believed responsible for the increased sensitivity of T-III to low temperature hydrogen embrittlement after aging for 1000 hours or longer near 1040°C. A total of eight Ta base alloys and two Cb base alloys were investigated. The effects of pre-age annealing temperature, aging time, temperature and environment, and alloy composition on the susceptibility to hydrogen embrittlement were investigated. The primary method of determining the effects of these variables on the ductility of T-III was by bend testing at 25° and -196°C. Fractured specimens were examined by the scanning electron microscope, electron microprobe, metallography and X-ray diffraction.

Alloy nominal composition,		СЪ	W	Hf	2r	Other	С	н	N	0
weight percent		Cont	ent, we	ight p	ercent		Co	ntent	, pp	n
Ta-10 W Ta-2 Hf Ta-8 W-0.5 Hf Ta-8 W-1 Hf Ta-8 W-2 Hf (T-111) Ta-8 W-3 Hf Ta-4 W-2 Hf Ta-8 W-1 Re-0.7 Hf-0.025 C (ASTAR 811C) Cb-1 Zr Cb-10 Hf-0.9 Ti-0.5 Zr- 0.4 Ta-0.4 W-0.015 C	Bal.	0.04 .04 .05 .04 .04 ———————————————————————————————	10.35 7.8 8.0 8.5 8.2 3.8 7.5 0.4	1.9 .51 .9 2.0 2.8 1.8 .65	0.02 .03 .06 .09 .06 	1.0 Re	15 22 50 50 50 60 50 260	1.0 1.0 1.6 2.9 1.0 0.8 3.0	40 39 9 5 15 8 7 32 70 40	21 33 30 24 120 28 26 25 73 130
(C103)	♦				l	l				

EXPERIMENTAL PROCEDURE

The chemical analyses of the alloys investigated in this study are given in Table I. The primary alloy under investigation in this program, T-111, was purchased in the form of sheet having a thickness of 0.8 mm and in the form of tubing having a 19 mm outside diameter and a 0.8 mm wall thickness. The remaining alloys were custom melted and fabricated to 0.8 mm thick sheet. Longitudinal bend specimens 6.4 mm X 25.4 mm were cut from the alloys. A standard annealing treatment for the Ta alloys consisted of 1hour at 1650°C plus 1-hour at 1315°C. The Cb alloys were annealed? at equivalent homologous temperatures, 1345° and 1070°C, respectively. In order to determine the effects of annealing temperature on subsequent hydrogen embrittlement, T-111 specimens were also annealed for 1-hour at 1815 or 1980°C plus 1-hour at 1315°C. A standard aging treatment for all ten alloys consisted of heating at 1040°C (the temperature which was associated with previous hydrogen embrittlement) for 1000 hours. One half of the specimens from each allow were sealed in a T-lll capsule containing lithium while the remaining half of the specimens were tied to the outside: of the capsule and heated in an ultrahigh vacuum of 1.3 X 10^{-/} . In addition a series of T-111 specimens were aged for 1000 hours at 925°C and a second series at 1150°C in order to bracket the primary aging temperature of 1040°C. A final series of T-111 specimens was aged for 5000 hours at 1040°C to determine the effect of aging time on subsequent hydrogen embrittlement.

After aging, removal of the lithium from the capsules was achieved by vacuum distillation or by dissolution in liquid ammonia. Several specimens from each alloy were then doped with approximately 10 ppm hydrogen by heating at 1040° C in a partial pressure of hydrogen of approximately 13 KN/m² for 10 minutes.

Primary evaluation of the alloy specimens was by means of bend testing at 25° and -196°C. Specimens were tested in a screw driven testing machine at a punch rate of 25.4 mm per minute. A bend radius of 2t and a total bend angle of about 160° was used for all the tests. After completing the bend test, specimens were then flattened so that in effect the tests were a 180°, 0t bend. Specimens were tested in the annealed and aged conditions before and after hydrogen doping.

After testing fracture surfaces were observed by scanning electron microscopy utilizing characteristic X-ray analysis to identify particles present on the fractured surface. In addition particles were identified by use of the electron microprobe and by X-ray diffraction of extracted particles. Standard light microscopy, transmission electron microscopy and scanning electron microscopy were utilized to characterize the microstructures of the alloys.

To further characterize the effects of aging on T-111 several specimens having different aging histories were also examined metallographically. Their aging conditions will be given at the appropriate place in the Results section.

RESULTS

Bend Tests

The effects of various annealing and aging conditions on the bend ductility of T-111 sheet and tubing are summarized in Table II. Specimens in the annealed condition or after aging at 925° and 1150°C underwent a full 180° - 0t bend at -196°C. All specimens aged at 1040°C independent of pre-age annealing temperature or aging time were brittle at -196°C. Testing tube specimens at 25°C resulted in ductile behavior for all conditions except for the specimen that had been annealed at 1980°C prior to aging. These results show that T-111 is susceptible to aging embrittlement over a narrow temperature range near 1040°C.

The effect of hydrogen doping on T-111 sheet and tubing is also summarized in Table II. Sheet T-111 in the annealed condition could undergo the full 180° - Ot bend test; however, surface cracks were observed on the specimen. A sheet specimen aged at 925°C could be bent 90° at -196°C prior to failure while a specimen aged at 1150°C was brittle upon testing at -196°C. The remaining sheet specimens and all tube specimens doped with approximately 10 ppm hydrogen were brittle at 25°C. The tube specimens appear to be more susceptible to hydrogen embrittlement, in agreement with previous results (2) where it was observed that aged tube samples were more susceptible than aged sheet samples

TABLE II. - EFFECT OF AGING CONDITIONS AND HYDROGEN DOPING ON

BEND DUCTILITY OF T-111 (Ta-8 W-2 Hf)

	1 -	T-	1				-		_				1	_			-	_	_	
test	Results		Ductile	Duct11e	Brittle	Ductile	Brittle	Ductile	Brittle	Ductile	Brittle	Brittle			Brittle	Brittle	Brittle	Brittle	Brittle	Brittle
Bend	Tempera- ture,		-196	-196	-196	25	-196	25	-196	25	-196	25] -	3	25	25	25	25	25	25
tons	Environ- ment	Tube	Vacuum	Lithium	Vacuum	Vacuum	Lithium	Lithium	Lithium	Lithium	Lithium	Lithium	Tube - Hudrogen Doned	ישפרו ישקרי	Vacuum	Lithium	Lithium	Vacuum	Lithium	Vacuum
Aging conditions	Tempera- ture, oc	Tu	925	1150	1040	1040	1040	1040	1040	1040	1040	1040	Tube - Hord		925	1150	1040	1040	1040	1040
Ag	Time,		1000	1000	2000	2000	1000	1000	1000	1000	1000	1000			1000	1000	2000	1000	1000	1000
Annealing	ture,		1650	1650	1650	1650	1650	1650	1815	1815	1980	1980			1650	1650	1650	1650	1815	1980
test	Results		Ductile	Duct11e	Ductile	Brittle	Brittle	Brittle	Brittle			1	oracks oo hard	Brittle	Brittle	Brittle	Brittle	Brittle		
Bend	Tempera- ture, oc		-196	-196	-196	-196	-196	-196	-196		pa	106	-196	-196	25	25	25	25		
tons	Environ- ment	Sheet		Lithium	Vacuum	Lithium	Vacuum	Lithium	Vacuum		- Hydrogen Doped		Vacuum	Lithium	Vacuum	Lithium	Vacuum	Lithium		
Aging conditions	Tempera- ture, oc	Sh		925	1150	1040	1040	1040	1040		Sheet - Hy		925	1150	1040	1040	1040	1040		
A	Time, hrs		-	1000	1000	2000	1000	1000	1000				1000	1000	5000	1000	1000	1000		
Annealing	ture,		1650	1650	1650	1650	1650	1815	1980			1650	1650	1650	1650	1650	1815	1980		

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TABLE 111. - EFFECT OF ALLOY COMPOSITION ON AGING EMBRITTLEMENT AND SUBSEQUENT SUSCEPTIBILITY TO HYDROGEN EMBRITTLEMENT

Condition	Hydrogen doped	Bend test		Condition	Hydrogen doped	Bend test					
	aopea	Tempera- ture, OC	Results		doped	Tempera- ture, oc	Results				
	Ta ~10	w		Ta-8 W-3 Hf							
Annealed Aged* Annealed Aged	No No Yes Yes	-196 -196 -196 -196	Ductile Ductile Ductile Cracks	Annealed Aged Aged Annealed Aged	No No No Yes Yes	-196 -196 25 -196 25	Ductile Brittle Ductile Ductile Brittle				
	Ta-2 1	lf		Ta-4 W-2 Hf							
Annealed Aged Annealed Aged	No No Yes Yes	-196 -196 -196 -196	Ductile Ductile Ductile Ductile	Annealed Aged Annealed Aged-lithium	No No Yes Yes	-196 -196 -196 25	Ductile Ductile Ductile Brittle				
	Ta-8 W-0.	5 Hf		Aged-vacuum	Yes	25	Cracks				
Annealed Aged Annealed Aged-lithium	No No Yes Yes	-196 -196 -196 25	Ductile Ductile Ductile Brittle	Ta-8 W-1 Re- Annealed Aged	No No	-196 -196	Ductile Ductile				
Aged-vacuum	Yes Ta-8 W-1	25 Hf	Cracks	Annealed Aged-lithium Aged-vacuum	Yes Yes Yes	-196 25 25	Ductile Brittle Cracks				
Annealed Aged	No No	-196 -196	Ductile Ductile		СЬ-1	Zr					
Annealed Aged-lithium Aged-vacuum	Yes	-196 25 25	Ductile Brittle Cracks	Annealed Aged Annealed	No No Yes	-196 -196 -196	Ductile Ductile Ductile				
Ta-8 W-2 Hf (T-111)				Aged Yes -196 Ductile Cb-10Hf-0.9Ti-0.5Zr-0.4Ta-0.4W (C103)							
Annealed Aged Aged Annealed	No No No Yes	-196 -196 25 -196 25	Ductile Brittle Ductile Cracks	Annealed Aged-vacuum Annealed	No No Yes	-196 -196 -196	Ductile Cracks Ductile				
Aged	Yes	1 23	Brittle	Aged-vacuum	Yes	-196	Cracks				

^{*}Aged 1000 hours at 1040° C.

to moisture in the bend test atmosphere. The results indicate that aging over the temperature range 925° to 1150°C increases the susceptibility to hydrogen embritlement of T-111 compared to the annealed condition

The effects of alloy composition on the aging and hydrogen embrittlement of Ta and Cb alloys are summarized in Table III. The results indicate that aging embrittlement in Ta alloys occurs only in T-111 (Ta-8W-2Hf) and the alloy Ta-8W-3Hf. The Cb alloy, Cl03 exhibited slight surface cracks in the aged condition which may be attributed to aging embrittlement. All the annealed alloys doped with hydrogen were ductile at -196°C with only the T-111 specimen exhibiting slight surface cracks as a result of the 180° -

Ot bend. With the exception of Ta-2Hf and Cb-1Zr all the aged alloys were susceptible to hydrogen embrittlement. The binary Ta alloy, Ta-10W, and the Cb alloy, Cl03, exhibited surface cracks when tested at -196°C. Tantalum alloys T-111 (Ta-8W-2Hf) and Ta-8W-3Hf were brittle at 25°C upon testing in the aged plus hydrogen doped condition. The remaining alloys that were aged in lithium prior to hydrogen doping were brittle at 25°C while those aged in vacuum underwent the 180° - Ot bend and exhibited only surface cracks. The reason for this ductility dependency on aging environment was shown to be related to a higher concentration of hydrogen in those specimens aged in lithium compared to those aged in vacuum. Oxygen analysis after aging showed that lithium removed oxygen from the modified T-III alloys while vacuum aging had no apparent effect on the oxygen content. Subsequent hydrogen doping under identical conditions showed a greater pickup of hydrogen in the low oxygen content (lithium aged) specimens (about 20 ppm compared to 10 ppm for vacuum aged) and hence the more brittle behavior at 25°C.

The results indicate the Ta and Cb binary alloys, especially Ta-2Hf and Cb-1Zr are not embrittled at -196°C as a result of the 1040°C aging plus hydrogen doping. In contrast the more complex aged Ta alloys are all embrittled by hydrogen when tested at 25°C while the complex Cb alloy, Cl03, suffered some loss of ductility at -196°C.

Metallography

The aging embrittlement and increased susceptibility to hydrogen embrittlement of aged Ta alloys is attributed to the change in microstructure upon long time aging near 1040°C. Aging T-111 for 1000 hours at 980°C results in formation of rows of precipitate particles lying along grain boundaries as shown in the transmission electron micrograph of figure 1(a). In contrast aging for 1000 hours at 1315°C results in a microstructure free of precipitates as shown in figure 1(b). This microstructure is characteristic of annealed T-III as well. Results of electron microprobe step-scan traverses along grain boundaries of annealed and aged T-111 are shown in figure 2. The annealed T-111 shows minor variations of Ta, W and O along grain boundaries; however, in the aged material, peaks of Hf and O were observed while the W remained constant. These results suggest the particles observed in figure 1 are Hf rich. X-ray diffraction results of particles remaining from dissolution of an aged T-111 specimen showed the residue to be HfO₂.

The effects of alloy composition of Ta alloys on fracture behavior and precipitate morphology are shown in figure 3. All specimens were aged 1000 hours at 1040°C, doped with hydrogen and

bend tested at 25°C. It should be noted that Ta-10W and Ta-2Hf. figure 3(a) and (b), fail in a ductile manner and are free of precipitate particles at grain boundaries. Alloys shown in figure 3(c) and (d), Ta-8W-.5Hf and Ta-8W-1Hf, respectively, are seen: to fail primarily in a brittle manner with some evidence of ductile failure. The Ta-8W-2Hf (T-111) and Ta-8W-3Hf alloys fracture in a completely brittle manner, as shown in figure 3(e) and (f). respectively. Precipitate particles are observed in all four of these ternary alloys with the amount of particle formation increasing with increasing Hf content. Use of an energy dispersive spectrometer on the scanning electron microscope showed that on the grain boundary surface in T-111 Hf could not be detected, figure 3(q), while the particles at the grain boundaries are Hf rich as shown in figure 3(h). Observations on the two Cb alloys showed that they failed in a ductile manner with no apparent formation of precipitates at grain boundaries

DISCUSSION

The aging and hydrogen embrittlement of T-111 and other similar Ta base alloys is believed to be due to Hf segregation at grain boundaries. The absence of particle formation in Ta-2Hf suggests that the presence of W in the ternary alloys affects the rate and degree of Hf segregation that is observed in these alloys. This may occur due to the lattice contraction that occurs upon adding W to Ta (3) causing the larger Hf atom to segregate to mifit or grain boundary areas. Competing with this equilibrium segregation process is diffusion which will tend to evenly disperse the solute at higher temperatures (4). Hence, T-111 aged at 1315°C did not exhibit precipitate particles as was shown in figure 1(b). Also this material did not exhibit aging embrittlement and was not susceptible to hydrogen embrittlement.

The role of Hf in Ta alloys and probably Zr or Hf in more complex Cb alloys in the hydrogen embrittlement problem is to segregate to grain boundaries during aging thus causing embrittlement at -196°C. Doping the aged material with hydrogen contributes an additional embrittling effect which is evident by the brittle behavior at 25°C of aged and hydrogen doped specimens. It appears that Hf segregation compounds the embrittlement caused by hydrogen since no embrittlement was observed in Ta-2Hf where Hf did not segregate. This increased embrittlement may arise due to Hf segregated at grain boundaries acting as a sink for hydrogen causing hydrogen to segregate at grain boundaries as well, in the ternary and more complex alloys.

For use as containment materials for alkali metals the ternary alloys Ta-8W-.5Hf, Ta-8W-1Hf, and Ta-4W-2Hf were not susceptible to aging embritlement, thus suggesting they are more attractive than

T-111 (Ta-8W-2Hf). These alloys were susceptible to hydrogen embrittlement after aging. In contrast Ta-2Hf was not susceptible to aging or hydrogen embrittlement and should also provide corrosion resistance. Results from this study indicate the tensile strength of Ta-2Hf is about 50 percent that of T-111 (Ta-8W-2Hf) at 1040°C which may limit its use at this temperature. The Cb alloys should also be considered as possible candidates based on their favorable ductility after aging and hydrogen doping.

CONCLUSIONS

Based on a study of the hydrogen embrittlement of aged Ta and Cb alloys the following conclusions are drawn:

- 1. Aging ternary Ta alloys such as T-III (Ta-8W-2Hf) near 1040°C for 1000 hours or longer increases their sensitivity to low temperature hydrogen embrittlement.
- 2. Segregation of Hf to grain boundaries during aging causes embrittlement upon testing at -196°C and is responsible for the observed hydrogen embrittlement.
- 3. Binary Ta and Cb alloys, Ta-2Hf and Cb-1Zr, are not susceptible to hydrogen embrittlement under the conditions of this study and did not exhibit grain boundary segregation of Hf or Zr.
- 4. Ternary alloys Ta-8W-.5Hf, Ta-8W-lHf, and Ta-4W-2Hf are superior to T-lll for containment of alkali metals in that they do not exhibit aging embrittlement. However, these alloys in the aged condition are susceptible to hydrogen embrittlement. Binary alloys Ta-2Hf and Cb-lZr are attractive containment materials based on retention of low temperature ductility after aging and hydrogen doping; however, their relatively low tensile strengths at 1040°C may limit their use.

REFERENCES

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TABLE I. - CHEMICAL ANALYSIS OF TANTALUM AND COLUMBIUM ALLOYS

Alloy nominal composition,	Ta	СР	W	Hf	Zr	Other	С	H	N	0
weight percent		Cont	ent, we	ight p	ercent		Со	ntent	, pp	m
Ta-10 W	Bal.		10.35				15	1.0	40	21
Ta-2 Hf	1			1.9			22	1.0	39	33
Ta-8 W-0.5 Hf		0.04	7.8	.51	0.02		50	1.0	9	30
Ta-8 W-1 Hf	11	.04	8.0	.9	.03	 	50	1.6	5	24
Ta-8 W-2 Hf (T-111)	11	.05	8.5	2.0	.06		50	2.9	15	120
Ta-8 W-3 Hf	1 1	.04	8.2	2.8	.09	 	60	1.0	8	28
Ta-4 W-2 Hf		.04	3.8	1.8	.06		50	0.8	7	26
Ta-8 W-1 Re-0.7 Hf-0.025 C	1 1		7.5	.65		1.0 Re	260	3.0	32	25
(ASTAR 811C)	\ ♦		1	i	}			l		1
Cb-1 Zr	`	Bal.			0.95		100	3.0	70	73
Cb-10 Hf-0.9 Ti-0.5 Zr-	0.4	1	0.4	9.8	.52	0.95 T1	<30	<5	40	130
0.4 Ta-0.4 W-0.015 C (C103)	↓									

TABLE II. - EFFECT OF AGING CONDITIONS AND HYDROGEN DOPING ON

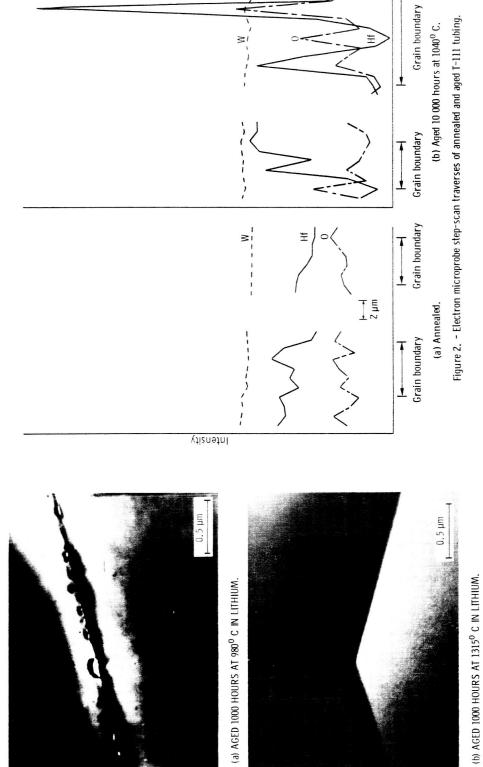
BEND DUCTILITY OF T-111 (Ta-8 W-2 Hf)

Environ- Tempera- Results Control Ture, Time, Tempera- Control	Ag		Aging conditions	ions	Bend test	test	Annealing	Ag	Aging conditions	lons	Bend	Bend test
Tube	Time, Tempera- hrs ture,	Tempera- ture, o _C		Environ- ment	Tempera- ture, O _C	Results	tempera- ture, O _C	Time, hr	Tempera- ture,	Environ- ment	Tempera- ture,	Results
1650 1000 925 Vacuum 1650 1000 1150 Lithium 1150 Lithi	Shee	Shee	9	ř.					Tul	be		
Lum -196 Ductile 1650 1000 1150 Lithium Lum -196 Brittle 1650 5000 1040 Vacuum Lum -196 Brittle 1650 1000 114hium Lum -196 Brittle 1650 1000 Lithium Lum -196 Brittle 1815 1000 1040 Lithium Lum -196 Brittle 1980 1000 1040 Lithium Lum -196 90° bend Lithium Lithium Lum -196 Brittle 1650 1000 1040 Lithium Lum -196 Brittle 1650 1000 1040 Lithium Lum 25 Brittle 1650 1000 1040 Lithium Lum 25 Brittle 1650 1000 1040 Vacuum Lum 25 Brittle 1650 1000 1040 Lithiu	-			1	-196	Ductile	1650	1000	925	Vacuum	-196	Duct11e
Tune -196 Ductile 1650 5000 1040 Vacuum 196 Brittle 1650 1000 1040 Vacuum 196 Brittle 1650 1000 1040 Lithium 196 Brittle 1815 1000 1040 Lithium 1980 1000 1040 Lithium 196 Brittle 1650 1000 1050 Lithium 1980 1000 1050 Lithium 1980 1000 1040 Vacuum 1980 1000 1000 1000 1000 1000 1000 1	1000 925 L		Н	1thium	-196	Duct11e	1650	1000	1150	Lithium	-196	Ductile
Lum -196 Brittle 1650 5000 1040 Vacuum Lum -196 Brittle 1650 1000 1040 Lithium 1 Doped Brittle 1815 1000 1040 Lithium 1 Doped Labe Lithium Lithium 1 Doped Labe Lithium 1 Doped Lithium Lithium <t< td=""><td>1150</td><td></td><td>5</td><td>acuum</td><td>-196</td><td>Ductile</td><td>1650</td><td>2000</td><td>1040</td><td>Vacuum</td><td>-196</td><td>Brittle</td></t<>	1150		5	acuum	-196	Ductile	1650	2000	1040	Vacuum	-196	Brittle
Tube 1650 1000 1040 14thium 196 Brittle 1650 1000 1040 14thium 14thium 196 Brittle 1815 1000 1040 14thium 14thium 1980 1000 1040 14thium 1980 1000 1040 14thium 1980 1000 1040 14thium 1980 1000 1040 14thium 196 Brittle 1650 1000 1050 14thium 196 Brittle 1650 1000 1040 14thium 1980 1000 1040 10	1040		Ä	[thium	-196	Brittle	1650	2000	1040	Vacuum	25	Duct11e
Lum -196 Brittle 1650 1000 1040 Lithium 1 1815 1000 1040 Lithium 1 1815 1000 1040 Lithium 1 1980 1000 1040 Lithium 1980 1000 1040 Lithium 11 1980 1000 Lithium 10 1000 1040 Lithium 10 Brittle 1650 1000 925 Vacuum 10 Brittle 1650 1000 1150 Lithium 10 Brittle 1650 1000 1040 Vacuum 10 25 Brittle 1650 1000 1040 Vacuum 10 25 Brittle 1650 1000 1040 Vacuum 10 1000 1040 Lithium	1040		S .	cuum	-196	Brittle	1650	1000	1040	Lithium	-196	Brittle
Tube	1040		검	thium	-196	Brittle	1650	1000	1040	Lithium	25	Ductile
1815 1000 1040 Lithium 1980 1000 1040 Lithium Lithium 1980 1000 1040 Lithium Lithium Lithium Libé 25 Brittle 1650 1000 1040 Lithium Lithium Lim 25 Brittle 1650 1000 1040 Lithium Lithium Lim 25 Brittle 1650 1000 1040 Lithium Lithium Lim 25 Brittle 1650 1000 1040 Lithium Lithium Libé Lithium Lithium Libé Lithium Lithium Libé Lithium L	1040		V.	cuum	-196	Brittle	1815	1000	1040	Lithium	-196	Brittle
Doped 1980 1000 1040 Lithium Lithi			1.				1815	1000	1040	Lithium	25	Ductile
-196 Cracks	Sheet - Hydrog	ŧ	drog	gen Dop	ed		1980	1000	1040	Lithium	-196	Brittle
-196 90° bend -196 Brittle 25 Brittle 25 Brittle 25 Brittle 25 Brittle 25 Brittle 26 Brittle 27 Door 1000 1150 Lithlum 28 Brittle 29 Door 1000 1150 Lithlum 29 Brittle 20 Doped 21 Doped 22 Dittlum 23 Dittlum 24 Doped 25 Brittle 26 Doped 27 Doped 28 Doped 28 Doped 29 Doped 20 Doped 21 Doped 21 Doped 22 Doped 23 Doped 24 Doped 25 Doped 26 Doped 27 Doped 28 Doped 28 Doped 28 Doped 29 Doped 20 Doped 2					-196	Cracks	1980	1000	1040	Lithium	25	Brittle
-196 Brittle 1650 1000 925 Vacuum 25 Brittle 1650 1000 1150 Lithium 25 Brittle 1650 1000 1040 Lithium 25 Brittle 1650 1000 1040 Vacuum 1815 1000 1040 Vacuum 1980 1000 1040 Vacuum	925	_	Vac	mnn	-196	90° bend			Tube - Hyd	rogen Dope	þ	
25 Brittle 1650 1000 925 Vacuum 25 Brittle 1650 1000 1150 Lithium 25 Brittle 1650 1000 1040 Lithium 25 Brittle 1650 1000 1040 Vacuum 1815 1000 1040 Vacuum 1980 1000 1040 Vacuum	1150	_	Lit	hfum	-196	Brittle						
25 Brittle 1650 1000 1150 Lithlum 25 Brittle 1650 5000 1040 Lithlum 25 Brittle 1650 1000 1040 Vacuum 1815 1000 1040 Vacuum 1980 1000 1040 Vacuum	5000 1040 Va		Va	cuum	25	Brittle		1000	925	Vacuum	25	Brittle
25 Brittle 1650 5000 1040 Lithium 25 Brittle 1650 1000 1040 Vacuum 1815 1000 1040 Lithium 1980 1000 1040 Vacuum	1040		;	thium	25	Brittle	1650	1000	1150	Lithium	25	Brittle
.um 25 Brittle 1650 1000 1040 Vacuum 1815 1000 1040 Lithium 1980 1000 1040 Vacuum	1040		S S	coun	25	Brittle	1650	2000	1040	Lithium	25	Brittle
1000 1040 Lithium 1000 1040 Vacuum	T040		3	thium	25	Brittle	1650	1000	1040	Vacuum	25	Brittle
1000 1040 Vacuum							1815	1000	1040	Lithium	25	Brittle
							1980	1000	1040	Vacuum	25	Brittle

TABLE III. - EFFECT OF ALLOY COMPOSITION ON AGING EMBRITTLEMENT AND SUBSEQUENT SUSCEPTIBILITY TO HYDROGEN EMBRITTLEMENT

Condition	Hydrogen	Bend	test	Condition	Hydrogen	Bend	test				
	doped	Tempera- ture, ^O C	Results		doped	Tempera- ture, OC	Results				
	Ta-10	W			Ta-8 W-3	Hf					
Annealed Aged* Annealed Aged	No No Yes Yes	-196 -196 -196 -196	Ductile Ductile Ductile Cracks	Annealed Aged Aged Annealed	No No No Yes	-196 -196 25 -196	Ductile Brittle Ductile Ductile				
Ta-2 Hf				Aged Yes 25 Brittle							
Annealed Aged Annealed Aged	No No Yes Yes	-196 -196 -196 -196	Ductile Ductile Ductile Ductile	Annealed Aged Annealed	No No Yes	-196 -196 -196	Ductile Ductile Ductile				
	Ta-8 W-0.5 Hf				Yes Yes	25 25	Brittle Cracks				
Annealed Aged	No No	-196 -196	Ductile Ductile	Ta-8 W-1 Re-	0.7 Hf-0.0	25 C (ASTA	R 811C)				
Annealed Aged-lithium Aged-vacuum	Yes Yes Yes Ta-8 W-1	-196 25 25	Ductile Brittle Cracks	Annealed Aged Annealed Aged-lithium Aged-vacuum	No No Yes Yes Yes	-196 -196 -196 25 25	Ductile Ductile Ductile Brittle Cracks				
Anna-1-4					Cb-1 Zr						
Aged Annealed Aged-lithium Aged-vacuum	No Yes Yes Yes	-196 -196 -196 25 25	Ductile Ductile Ductile Brittle Cracks	Annealed Aged Annealed Aged	No No No Yes Yes	-196 -196 -196 -196	Ductile Ductile Ductile Ductile				
	1-8 W-2 Hf	r		Cb-10Hf-0.9	Ti-0.5Zr-0	.4Ta-0.4W	(C103)				
Annealed Aged Aged Annealed Aged	No No No Yes Yes	-196 -196 25 -196 25	Ductile Brittle Ductile Cracks Brittle	Annealed Aged-vacuum Annealed Aged-vacuum	No No Yes Yes	-196 -196 -196 -196	Ductile Cracks Ductile Cracks				

^{*}Aged 1000 hours at 1040° C.



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Figure 1. - Transmission electron micrographs of aged T-111 sheet.

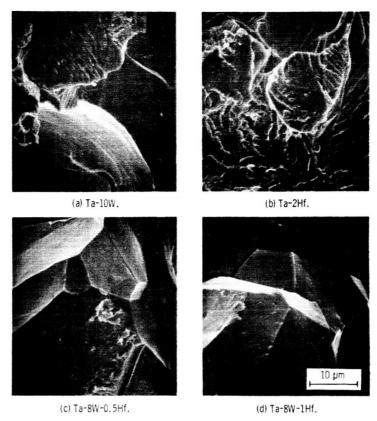


Figure 3. – Scanning electron microscope observations and analysis of precipitate particles in 1000 hour – 1040° C aged Ta alloys.

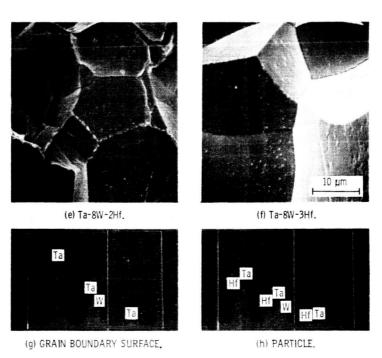


Figure 3. - Concluded.